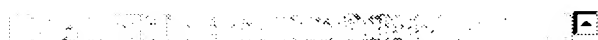


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USPT	116 and nanofiltration	16	<u>L17</u>
USPT	membrane and koch	1657	<u>L16</u>
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USPT	210 500 38	566	<u>L12</u>
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USPT	17 and koch	0	<u>L9</u>
USPT	17 and "SR1"	0	<u>L8</u>
USPT	TFC and NF and 11	9	<u>L7</u>
USPT	14 and nanofiltration	0	<u>L6</u>
USPT	14 and ananofiltration	0	<u>L5</u>
USPT	"SR1" and 11	20	<u>L4</u>
USPT	"TFC-SR1"	0	<u>L3</u>
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116 and nanofiltration

16





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1. Document ID: US 5858240 A

File: USPT

Jan 12, 1999

L11: Entry 1 of 3

US-PAT-NO: 5858240

DOCUMENT-IDENTIFIER: US 5858240 A

TITLE: Nanofiltration of concentrated aqueous salt solutions

DATE-ISSUED: January 12, 1999

INVENTOR INFORMATION:

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COUNTRY

CAX

CAX

US-CL-CURRENT 210/652; 210/639, 210/641, 210/651, 210/653, 210/805

Full	Title	Citation	Front	Review	Classification	Date	Reference	Claims	KWIC	Draw Desc	Image
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2. Document ID: US 5587083 A

File: USPT

Dec 24, 1996

L11: Entry 2 of 3

US-PAT-NO: 5587083

DOCUMENT-IDENTIFIER: US 5587083 A

TITLE: Nanofiltration of concentrated aqueous salt solutions

DATE-ISSUED: December 24, 1996

INVENTOR INFORMATION:

NAME

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CAX

US-CL-CURRENT: 210/652; 210/651, 210/653

Full	Title	Citation	Front	Review	Classification	Date	Reference	Claims	KWIC	Draw Desc	Image
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3. Document ID: US 5147553 A

File: USPT

Sep 18, 1997

L11: Entry 3 of 3

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111: Entry 1 of 3

File: USPT

Jan 12, 1997

DOCUMENT-IDENTIFIER: US 5898247 A

TITLE: Nanofiltration of concentrated aqueous salt solutions

ABST:

Nanofiltration processes using one or more conventional nanofiltration membrane modules under a positive applied pressure is used to selectively change the concentration of one solute, such as sodium chloride or sodium chlorate providing monovalent ions, from another solute such as sodium sulfate or sodium dichromate to provide multivalent ions in high salt aqueous concentrations. The process is particularly useful in favourably lowering the concentration of undesirable ions, particularly, of silica and dichromate ions in chloralkali and chlorate brine containing solutions and favourably raising the sodium sulphate level relative to sodium chloride in chloralkali liquor.

BSPR:

This invention relates to a process for reducing the concentration of undesirable compounds, particularly, solutes, in aqueous solutions by nanofiltration using a filtration membrane. More particularly, it relates to the substantial removal of sulfate, dichromate, phosphate, mercury, calcium, magnesium, aluminium, fluoride and silica monovalent and divalent ions from brine solutions, optionally, containing chlorate.

BSPR:

Pressure driven membrane separation processes are known wherein organic molecules or inorganic ionic solutes in aqueous solutions are concentrated or separated to various degrees by the application of a positive osmotic pressure to one side of a filtration membrane. Examples of such pressures are reverse osmosis (RO), ultrafiltration (UF) and nanofiltration (NF). These pressure driven membrane processes employ a cross-flow mode of operation wherein only a portion of a feed solution (F) is collected as a permeate solution (P) and the rest is collected as a pass solution (C). In this specification and claims, the exit process stream from the nanofiltration module, which stream has not passed through the membrane is referred to as the "pass stream". This stream is often referred to by practitioners in the membrane filtration art as the "concentrate" stream.

BSPR

Nanofiltration membranes are structurally very similar to reverse osmosis membranes in that chemically, they, typically, are crosslinked aromatic polyamides, which are cast as a thin "skin layer", on top of a microporous polymer sheet support to form a composite membrane structure. The separation properties of the membrane are controlled by the pore size and electrical charge of the "skin layer". Such a membrane structure is usually referred to as a thin film composite (TFC). However, unlike RO membranes, the NF membranes are characterized in having a larger pore size in its "skin layer" and a net negative electrical charge inside the individual pores. This negative charge is responsible for rejection of anionic species, according to the anion surface charge density. Accordingly, divalent anions, such as $\text{SO}_4^{\text{sup.}} \text{sup.}$, are more strongly rejected than monovalent ones, such as $\text{Cl}^{\text{sup.}}$. Commercial NF membranes are available from known suppliers of RO and other pressure drive membranes. Examples include: Desal-5 membrane (Desalination Systems, Escondido, Calif.), NF70, NF50, NF40 and NF40NF membranes (FilmTec Corp., Minneapolis, Minn.), SFC membranes (Toray, Japan), and NRT 7450 and NTR 7350 membranes (Nitto Electric, Japan). The NF membranes are, typically, packaged as membrane modules. A so-called "spiral wound" module is most popular, but other membrane configurations, such as tubular membranes enclosed in a shell of plate and frame type, are also known.

BSFR:

Nanofiltration membranes have been reported to show no or little rejection of low molecular weight organic molecules, such as, methanol, ethanol and ethyleneglycol, but a significant rejection of higher molecular weight organic species, such as glucose. Among inorganic ionic solutes, low molecular weight species has been reported for simple 1:1 electrolytes, such as NaCl or KCl, and a high rejection of other electrolytes where multivalent ionic species are involved, such as Na₂SO₄, MgCl₂ and FeCl₃. Such a characteristic differentiates NF from RO which rejects all ionic species, and from ultrafiltration UF, which does not reject ionic species and only rejects organic compounds with molecular weights, typically, in excess of 1,000 g/mol.

BSFR:

During the NF process, a minimum pressure equal to the osmotic pressure difference between the feed pass liquor on one side and the permeate liquor on the other side of the membrane must be applied since osmotic pressure is a function of the ionic strengths of the two streams. In the case of separation of a multivalent solute, such as Na₂SO₄, from a monovalent one, such as NaCl, the osmotic pressure difference is moderated by the low NaCl rejection. Usually, a pressure in excess of the osmotic pressure difference is employed to achieve practical permeate flux. In view of lower NaCl rejection, NF has been used successfully for removal of sulfate and the hardness cations, Ca²⁺ and Mg²⁺ from brackish waters and even seawater, without the necessity to excessively pressurize the feed stream. The reported typical pressure range for NF is 80 to 300 psi, although membrane elements are designed to withstand pressures of up to 1,000 psi.

BSFR:

Reported uses of NF include the aforesaid water softening, removal of dissolved multivalent ions such as Fe²⁺, reduction of silica as a part of feedwater conditioning for a subsequent RO step or removal of medium molecular weight organic compounds. It has also been demonstrated that high rejection of ionic species could be obtained by proper conditioning of the stream, i.e. by changing its pH. Thus, effective removal (rejection) of carbonate anion could be achieved by adjusting the pH of the feed solution to about 12, to ensure that carbonate would predominantly exist as CO₃⁼, which anion is more strongly rejected by the NF membrane than the HCO₃⁻ anion.

BSFR:

Dissolved or suspended silica in brine feed for chloralkali processes, especially the so-called membrane chloralkali process, presents a problem in that the silica forms scale on the surface or in the interior of the ion exchange membrane separator. This causes the cell voltage and, hence, power consumption to increase. In general, in the membrane chloralkali process, the concentration of silica in the feed brine should not exceed 10 ppm, although even a lower level may be needed if some other contaminants, such as Al³⁺, are present, since these contaminants enhance the scaling capacity of silica.

BSFR:

Use of strongly basic anion exchange membranes for silica removal from feedwater has been reported. However, the literature also recognizes that, in that case since there is a substantial background of other salts, the selectivity of the IX resin towards silica is greatly reduced.

BSFR:

Product literature from FilmTec Corp., Minneapolis, Minn., describes the removal of silica from feedwater with a NF70 nanofiltration membrane as part of a pretreatment for a subsequent RO step. A rejection of 95% silica concentration in feedwater from 400 ppm to 50-60 ppm has been mentioned. The literature is silent, however, on the use of NF methods for silica removal from higher concentration salt solutions, such as chloralkali brine.

BSFR:

In a related chemical process, chlorine and caustic soda are prepared in an electrolytic cell, which contains a membrane to prevent chlorine and caustic from reacting and the separated chemicals are removed.

BSFR:

sulfate ion is a common ingredient in commercial salt. When such salt is used directly, or in the form of a brine solution, and specific steps are not taken to remove the sulfate, the sulfate enters the electrolytic system. Sulfate ion maintains its identity under the conditions in the electrolytic system and, thus, accumulates and progressively increases in concentration in the system unless removed in some manner. In chlorate plants producing a liquor product, the sulfate ion will leave with the product liquor. In plants producing only crystalline chlorate, the sulfate remains in the mother liquor after the crystallization of the chlorate, and is recycled to the cells. Over time, the concentration of sulfate ion will increase and adversely affect electrolysis and cause operational problems due to localized precipitation in the electrolytic cells. Within the chloralkali circuit, the sodium sulfate will concentrate and adversely effect the membrane, which divides the anolyte brine from the catholyte caustic soda.

BSIR:

Accordingly, in its broadest aspect the invention provides in a nanofiltration process for filtering an aqueous liquor comprising feeding a feed liquor to a nanofiltration membrane module under a positive pressure to provide a pass liquor and a permeate liquor for selectively changing the concentration of a first compound relative to the concentration of a second compound in said aqueous liquor wherein said first compound has a first feed concentration and said second compound has a second feed concentration, said process comprising feeding said aqueous liquor to said nanofiltration membrane module, collecting said pass liquor wherein said first compound is at a first pass concentration and said second compound is at a second pass concentration, and collecting said permeate liquor wherein said first compound has a first permeate concentration and said second compound is at a second permeate concentration, the improvement comprising said first compound having a first concentration of greater than 50 g/l.

BSPR:

We have discovered that, for example, subjecting a concentrated sodium chloride-sodium sulfate solution to the NE process results in a very strong positive rejection of sodium sulfate by the membrane, but only a weak positive to negative rejection of the sodium chloride. Surprisingly, we have found that with increasing concentration of sodium sulfate in the feed liquor, the rejection of sodium chloride decreases and eventually becomes negative. However, sodium chloride rejection need not be negative in that the NaCl concentration in the permeate may be slightly lower than that in the feed. The pronounced influence of Na.sub.2 SO.sub.4 concentration in the feed on NaCl rejection values is most surprising.

BSPR:

Thus, surprisingly we have found that nanofiltration membrane processes can be used to beneficially reduce the concentration of multivalent ions, such as SO.sub.4.sup.=, CrO.sub.4.sup.= or Cr.sub.2 O.sub.7.sup.= and dissolved silica in concentrated solutions of sodium chloride, such as brine, and concentrated sodium chlorate process liquors, wherein the main components are sodium chlorate and sodium chloride.

BSPR:

We have most surprisingly found, notwithstanding the teachings that commercially available nanofiltration membranes have a monocharged anion rejection property, e.g. a Cl.sup.- ion rejection in the range 20-50%, that such membranes when used with concentrated salt solutions exhibit no Cl.sup.- ion rejection. This unexpected absence of chloride rejection by the nanofiltration membrane has a significant practical importance in minimizing the osmotic pressure across the membrane and hence the energy required for pressuring the feed to achieve a high permeate flow. Further, in surprising contrast, the rejection of multivalent ions such as SO.sub.4.sup.=, CrO.sub.4.sup.= or Cr.sub.2 O.sub.7.sup.= and, also, silica, remains high.

BSPR:

Accordingly, such unexpected ion membrane selectivity at relatively high salt concentrations offers attractive applications such as, for example, in the treatment of chloralkali brine liquors having sodium sulfate levels unacceptable in recycle systems. As illustrated in an application of sulfate removal from brine, because there is no buildup in concentration of sodium chloride in the pass liquor stream over its original level in the feed stream, it is possible

increase the content of sodium sulfate in the pass liquor to a higher level than would have been possible if the NaCl level of the pass liquor was increased. Accordingly, it is now possible to realize a desirable high % Recovery, and, in the case of chloralkali brine, to minimize the volume of waste liquor and, in the case of a reactor and the amount of chemicals for an optional subsequent sulfate precipitation step.

BSIR:

The processes of the invention are applicable as either single stage unit, processes with optional recycle of either pass liquor or permeate liquor to the nanofiltration membrane module, or as part of a multi-stage, multi-module system.

BSPV:

The process of the invention as hereinabove defined may be operated at any suitable and desired temperature selected from -1 degree C. to the boiling point of the feed liquor; and positive pressures applied to the feed side, generally selected from 50-1200 psi.

BSPV:

(a) Desal-5 Membrane Product Application Note, publication of Desalination Systems, Inc. (Escondido, Calif.), April 1991, wherein the Figure on page E-19.3 shows NaCl rejection in the 85 to 88% range.

BSPV:

(b) NT70 Membrane, Product Specification, publication of Filmed Corp. Minneapolis, Minn., cites Rejection of 60%; and

BSPV:

(c) "Membrane Handbook", ed. By W. S. W. Ho and K. K. Sirkar, Van Nostrand Reinhold, N.Y. 1992 at Table 23.2. "Characteristics of Selected Nanofiltration Membranes", cites NaCl % Rejection of: 80% for NF50 membrane (Filmed), 48% for NF40 membrane (Filmed), 50% for NTF-720 membrane (Nitto), 47% for Desal-5 membrane (Desalination Systems), and 55% for SU200HF membrane (Toray).

DEPR:

FIG. 1 represents a diagrammatic flow sheet of a single stage membrane nanofiltration system of use in a process according to the invention; and

DEPR:

FIG. 2 represents a diagrammatic flow sheet of a multistage membrane nanofiltration system of use in a process according to the invention.

DEPR:

FIG. 1 shows generally as 10, a single stage membrane nanofiltration system for the separation of, for example, solute A from solute B in an aqueous liquor. System 10 comprises a feed solution holding tank 12 connected to a nanofiltration membrane module 14 by a feed conduit 16 through a high pressure pump 18 (Model I-2401, CIB Pumps Inc.). Module 15 comprises a single spiral wound type nanofiltration module containing Desal-5, DL2540 polyamide membrane 20 having 2.5 m.sq.2 of active membrane area. Exiting module 14 is a pass liquor conduit 22 having a pressure control valve 24 and a permeate liquor conduit 26.

DEPR:

The process depicted in FIG. 1 represents a single stage or batch-type process, wherein the pass liquor or the permeate liquor may be of sufficient and desired quality for use in a subsequent process or discharge. However, each of the pass and permeate streams, optionally, individually, may be sent through a nanofiltration membrane process again, in one or more cycles. In either a batch or continuous process. In industrial processes of use in the practice of the invention, the pass stream from the first stage may be sent to the second stage to increase the overall % Recovery. Alternatively, the NF process may be conducted in a batch mode with the pass liquor recycled back to the feed tank. Accordingly, in consequence, the feed composition will vary with time as will the Membrane Flux and possibly the % Rejection.

DEPR:

FIG. 2 represents a multi-stage NF method for the purification of brine by removal of sodium sulfate, according to the invention, wherein a plurality of NF

modules 14A-14F according to the invention, six in the first invention and five in the second invention, are connected in series. Feed liquor is fed under pressure by high pressure pump 5, to module 14A. Pass liquors 14B-14F are pumped to subsequent adjacent modules, while the permeate liquors 14A-14F may be combined into a single resultant purified brine stream. The final pass liquor may be discharged to sewer.

DEPR:

The following Examples illustrate specific compounds used in the process as described by FIG. 1 wherein data was collected using an experimental NF test cell which consisted of a single NF membrane filter element, 1.5" diameter, 2' long, containing Desal-5 DL2541 membrane from Desalination Systems, Inc., Escondido, Calif. The active membrane area was 2.5 m² sup. 2. All runs were conducted at temperature 45.degree.-50.degree. C.

DEPR:

A batch of 80 liters of brine solution containing 190.1 gpl NaCl and 18.3 gpl Na.sub.2 SO.sub.4 at a temperature of 45.degree. C. was added to tank 12. High pressure pump 18 was turned on and the pressure on the feed side was adjusted to 400.-1.5 psi and kept constant during the run. Both permeate and pass liquor streams were collected into separate flow tanks over a period of 11.5 minutes. Both the permeate and the pass liquor flow rates were approximately constant during the run at about 2.0 lpm and 3.3 lpm, respectively. Following the run, 25 l of permeate with a composition of 190.1 gpl NaCl and 0.25 gpl Na.sub.2 SO.sub.4 and 34 l of concentrate with a composition of 190.7 gpl NaCl and 18.3 gpl Na.sub.2 SO.sub.4 were collected while 20 l of the feed brine remained in the feed tank. Calculated NF membrane % Rejections are: 97.3% for Na.sub.2 SO.sub.4 and 1.1% for NaCl.

DEPR:

A similar process was carried out under the same conditions as for Example 1, wherein the volume of feed brine was 76 liters containing 195.9 gpl NaCl and 18.3 gpl Na.sub.2 SO.sub.4. After 10 minutes of operation with a feed pass pressure maintained at 400.-1.5 psi, 18 l of permeate and 3 l of concentrate were collected while 19 l of the feed brine remained in the feed tank. The composition of the permeate was 194.7 gpl NaCl and 0.37 gpl Na.sub.2 SO.sub.4. The composition of concentrate was 192.0 gpl NaCl and 26.3 gpl Na.sub.2 SO.sub.4. Calculated NF membrane % Rejections are: 97.9% for Na.sub.2 SO.sub.4 and 1.4% for NaCl.

DEPR:

In this example a recycle batch mode of operation was carried out wherein the pass stream was recycled back to the brine feed tank. A starting volume of feed brine was 76 l having a composition of 197.5 gpl NaCl and 16.7 gpl Na.sub.2 SO.sub.4. The flow rate of permeate was kept constant at 1.3 lpm. The resulting feed pass pressure was initially at 295 psi and at the end of the process at 315 psi. After 45.5 minutes 50 l of permeate were collected while the volume in the brine feed tank decreased to 26 l. The composition of permeate was 200.4 gpl NaCl and 0.38 gpl Na.sub.2 SO.sub.4. The composition of brine solution remaining in the feed tank was 188.4 gpl NaCl and 44.8 gpl Na.sub.2 SO.sub.4. The calculated NF membrane % Rejections were 97.7% for Na.sub.2 SO.sub.4 and -1.5% for NaCl.

DEPR:

A recycle batch process similar to that of Example 3 was carried out with a liquor further containing silica and having a pH of 10.7. The initial volume of brine feed solution was 78 l and had a composition of 246.9 gpl NaCl, 30.1 gpl Na.sub.2 SO.sub.4 and 9.1 ppm SiO.sub.2. The feed pass liquor pressure was kept constant at 320.-1.5 psi. After 64 minutes 50 l of permeate was collected while the volume of solution in the feed tank decreased to 28 l. The composition of permeate was 257.5 gpl NaCl, 3.85 gpl Na.sub.2 SO.sub.4 and 5.6 ppm SiO.sub.2. The composition of brine solution remaining in the feed tank was 241.5 gpl NaCl, 79.8 gpl Na.sub.2 SO.sub.4 and 15.1 ppm SiO.sub.2. The calculated NF membrane % Rejections were 97.1% for Na.sub.2 SO.sub.4, 9.3% for NaCl and 39.6% for SiO.sub.2.

DEPR:

A batch of 75 l of chlorate liquor feed solution containing 195 gpl NaClO.sub.3, 1.1 gpl NaCl, 20.8 gpl Na.sub.2 SO.sub.4, 5.1 gpl Na.sub.2 Cr.sub.2 O.sub.7 at a pH of 7 and a temperature of 45.degree. C. was added to tank 12. The high

pressure in the feed side was adjusted to 44.1 psi and kept constant during the run. Permeate and pass liquor streams were collected and analyzed over a period of 30 minutes. In total, 1.2 l of permeate liquor and 1.1 l of liquor were collected, while at 44.1 psi feed solution retained in the rig at the end of the run. The calculated average permeate liquor and concentrate flows were 0.02 lpm and 0.11 lpm, respectively. Surrogate chemical analysis revealed that the permeate liquor had 194 gpl NaCl, 101.3 gpl Na₂SO₄, 4.1 gpl Na₂SO₄ SO₄ sub.4, and 2.1 gpl Na₂SO₄ SO₄ sub.7, while the pass stream had 291 gpl NaCl, 101.3 gpl Na₂SO₄, 4.1 gpl Na₂SO₄ SO₄ sub.4, and 2.1 gpl Na₂SO₄ SO₄ sub.7. Calculated NF membrane % Rejections were: 99.8% for NaCl, 99.8% for Na₂SO₄ SO₄ sub.4, 99.8% for Na₂SO₄ SO₄ sub.7, and 99.8% for Na₂SO₄ SO₄ sub.7.

DEPR:
The following examples 6-9 further demonstrate the range of NaCl rejection during NF membrane treatment of NaCl-Na₂SO₄ SO₄ sub.4 solutions.

DEPR:
A batch of 80 liters of feed brine solution composed of 189.2 g/l NaCl and 11.4 g/l of Na₂SO₄ SO₄ sub.4 was passed to the NF rig. Under an applied pressure of 288 psi, 10 liters of permeate liquor were collected, which represents 12.8% recovery. The permeate had a composition of 177.9 g/l NaCl and 0.86 g/l Na₂SO₄ SO₄ sub.4. The calculated rejections were: 99.8% for NaCl and 99.8% for Na₂SO₄ SO₄ sub.4, respectively.

DEPR:
A batch of 80 liters of feed brine solution composed of 184.2 g/l NaCl and 11.4 g/l of Na₂SO₄ SO₄ sub.4 was passed to the NF rig. Under an applied pressure of 302 psi, 28 liters of permeate liquor were collected, which represents 38% recovery. The permeate had a composition of 186.7 g/l NaCl and 0.2 g/l Na₂SO₄ SO₄ sub.4. The calculated rejections were: 99.8% for NaCl and 99.8% for Na₂SO₄ SO₄ sub.4, respectively.

DEPR:
A batch of 80 liters of feed brine solution composed of 179 g/l NaCl and 11.3 g/l of Na₂SO₄ SO₄ sub.4 was passed to the NF rig. Under an applied pressure of 348 psi, 9 liters of permeate liquor were collected, which represents 11.2% recovery. The permeate had a composition of 196.0 g/l NaCl and 0.9 g/l Na₂SO₄ SO₄ sub.4. The calculated rejections were: 99.8% for NaCl and 99.8% for Na₂SO₄ SO₄ sub.4, respectively.

DEPR:
A batch of 80 liters of feed brine solution composed of 9.9 g/l NaCl and 87.5 g/l of Na₂SO₄ SO₄ sub.4 was passed to the NF rig. Under an applied pressure of 517 psi, 9.6 liters of permeate liquor were collected, which represents 12% recovery. The permeate had a composition of 18.4 g/l NaCl and 2.4 g/l Na₂SO₄ SO₄ sub.4. The calculated rejections were: 99.8% for NaCl and 99.8% for Na₂SO₄ SO₄ sub.4, respectively.

DEPR:
A batch of 80 liters of feed brine solution having a NaCl concentration of 28.8 g/l, pH 3, and spiked with 2.9 ppm F₂sup.- was passed to the NF rig. Under an applied pressure of 501 psi, 8.6 liters of permeate liquor were collected, which represents 10.7% recovery. The permeate had a composition of 281 g/l NaCl and 2.9 ppm F₂sup.-. The calculated rejections were: 99.8% for NaCl and 99.8% for F₂sup.-, respectively.

DEPR:
A batch of 80 liters of feed brine solution having a NaCl concentration of 28.8 g/l, pH 3, spiked with 3.2 ppm F₂sup.- and 1.5 ppm Fe₂sup.+, was passed to the NF rig. Under an applied pressure of 544 psi, 8.9 liters of permeate liquor were collected, which represents 11% recovery. The permeate had a composition of 281 g/l NaCl and 2.9 ppm F₂sup.-. The calculated rejections were: 99.8% for NaCl and 99.8% for F₂sup.-, respectively.

DEPR:
A batch of 80 liters of feed brine solution having a NaCl concentration of 28.8 g/l, pH 3, spiked with 3.1 ppm F₂sup.- and 2 ppm Fe₂sup.+, was fed to the NF rig. Under an applied pressure of 554 psi, 8.9 liters of permeate liquor were

collected which represents 1.5% recovery. The permeate contained 1.0 g/l NaCl and 0.1 ppm Fe.supp.+. The calculated rejections were 99.9% for NaCl and 99.9% for Fe.supp.+, respectively.

DEPR:

A batch of 80 liters of feed sulphate brine solution containing 4.0 g/l NaCl and 0.0003 and 0.0004 g/l NaCl at a pH 1.5, was passed to the NF rig. Under an applied pressure of 575 psi, 7.0 liters of permeate liquor were collected which represents 8.8% recovery. The permeate contained 1 g/l NaCl and 0.0003 and 0.0004 g/l NaCl. The calculated rejections were 99.9% for NaCl and 99.9% for NaCl, respectively.

DEPR:

A batch of 80 liters of feed sulphate brine solution containing 4.0 g/l NaCl and 0.0003 and 0.0004 g/l NaCl at a pH 1.5 was passed to the NF rig. Under an applied pressure of 575 psi, 7.0 liters of permeate liquor were collected which represents 8.8% recovery. The permeate contained 1 g/l NaCl and 0.0003 and 0.0004 g/l NaCl. The calculated rejections were 99.9% for NaCl and 99.9% for NaCl, respectively.

DEPR:

This example demonstrates the efficacy of the NF method for removal of Al.supp.+ from brine.

DEPR:

A batch of 80 liters of feed brine solution containing 249.2 g/l NaCl and 1.5 ppm Al.supp.+ was passed to the NF rig. Under an applied pressure of 461 psi, 24.6 liters of permeate liquor were collected, which represents 30% recovery. The permeate contained 245.7 g/l NaCl and about 1 ppm Al.supp.+. The calculated rejections were: 1.4% for NaCl and 90.5% for Al.supp.+, respectively.

DEPR:

This example demonstrates the efficacy of the NF process for the removal of mercury from brine.

DEPR:

A batch of 80 liters of feed brine containing 249.2 g/l NaCl and 1.5 ppm Hg.supp.++ was passed to the NF rig. Under an applied pressure of 465 psi, 24.6 liters of permeate liquor were collected, which represents 30.6% recovery. The permeate contained 244.8 g/l NaCl and 7.1 ppm Hg.supp.++. The calculated rejections were: 1.8% for NaCl and 29% for Hg.supp.++, respectively.

DEPR:

Examples 17-20 demonstrate the efficacy of the NF method for the removal of Ca.supp.++ and Mg.supp.++ from brine and the effect of pH and brine strength thereon.

DEPR:

A batch of 80 liters of feed brine containing 288.7 g/l NaCl and 100 ppm Ca.supp.++ at a pH of 7.8 was passed to the NF rig. Under an applied pressure of 295 psi, 9.5 liters of permeate liquor were collected, which represents 11.9% recovery. The permeate contained 282.5 g/l NaCl and 87.5 ppm Ca.supp.++. The calculated rejections were: 2.3% for NaCl and 12.5% for Ca.supp.++, respectively.

DEPR:

A batch of 80 liters of feed brine containing 288.7 g/l NaCl and 100 ppm Ca.supp.++ at a pH of 2.0 was passed to the NF rig. Under an applied pressure of 301 psi, 9.5 liters of permeate liquor were collected, which represents 11.9% recovery. The permeate contained 285 g/l NaCl and 87.5 ppm Ca.supp.++. The calculated rejections were: 2.2% for NaCl and 29% for Ca.supp.++, respectively.

DEPR:

A batch of 80 liters of feed brine containing 288.7 g/l NaCl and 100 ppm Ca.supp.++ at a pH of 2.0 was passed to the NF rig. Under an applied pressure of 301 psi, 9.5 liters of permeate liquor were collected, which represents 11.9% recovery. The permeate contained 285 g/l NaCl and 87.5 ppm Ca.supp.++. The calculated rejections were: 2.2% for NaCl and 29% for Ca.supp.++, respectively.

DEPR:

Example 1: A batch of 40 liters of feed brine containing 225 g/l NaCl and 10 g/l K.sub.2SO.sub.4 was passed to an NF rig. Under an applied pressure of 20 p.s.i., 14 liters of permeate liquor were collected, which represents 17.5% recovery. The permeate contained 21.5 g/l NaCl and 0.4 g/l K.sub.2SO.sub.4. The calculated rejections were: 2.9% for NaCl and 96.2% for K.sub.2SO.sub.4, respectively.

EXAMPLE:

Examples 21 and 22 demonstrate the efficacy of the NF treatment with respect to the separation of K.sub.2SO.sub.4 from KCl and RBr brines.

EXAMPLE:

A batch of 40 liters of feed brine containing 225 g/l KCl and 10 g/l K.sub.2SO.sub.4 was passed to the NF rig. Under an applied pressure of 20 p.s.i., 14 liters of permeate liquor were collected, which represents 17.5% recovery. The permeate contained 21.5 g/l KCl and 0.4 g/l K.sub.2SO.sub.4. The calculated rejections were: 2.9% for KCl and 96.2% for K.sub.2SO.sub.4, respectively.

EXAMPLE:

A batch of 40 liters of feed brine containing 225 g/l KBr and 10 g/l K.sub.2SO.sub.4 was passed to an NF rig. Under an applied pressure of 20 p.s.i., 14 liters of permeate liquor were collected, which represents 17.5% recovery. The permeate contained 21.5 g/l KBr and 0.4 g/l K.sub.2SO.sub.4. The calculated rejections were: 4.2% for KBr and 97.1% for K.sub.2SO.sub.4, respectively.

CLAIM:

1. In a nanofiltration process for filtering liquor comprising feeding feed liquor to a nanofiltration membrane module under a positive applied pressure to provide a pass liquor and a permeate liquor for selectively changing the concentration of a first compound relative to the concentration of a second compound in said aqueous liquor, wherein said first compound has a first feed concentration and said second compound has a second feed concentration, the process comprising feeding said aqueous liquor to said nanofiltration membrane module, collecting said pass liquor wherein said first compound is at a first pass concentration and said second compound is at a second pass concentration, and collecting said permeate liquor wherein said first compound has a first permeate concentration and said second compound is at a second permeate concentration, the improvement comprising said first compound having a first feed concentration of greater than 50 g/L; the ratio of the first feed compound concentration to the second feed compound concentration in the permeate liquor increases and in the pass liquor decreases relative to their ratio in the feed liquor; and wherein said first feed compound contains polyvalent ions and said second feed compound contains divalent trivalent ions.

CLAIM:

9. A process as defined in claim 1 further comprising feeding said pass liquor to one or more nanofiltration membrane modules connected in series so as to produce a second pass liquor and subsequent pass liquors, consecutively.

CLAIM:

10. A process as defined in claim 1 further comprising feeding said permeate liquor to one or more nanofiltration membrane modules connected in series so as to produce a second permeate liquor and subsequent permeate liquors, consecutively.

CLAIM:

11. A process as defined in any one of claims 1, 9 or 10, wherein said pass liquor or said permeate liquor is recycled back to one or more of said nanofiltration membrane modules.

REFERENCE:

Desalination System, Inc. Escondido, CA, Apr. 1991, "Desal-1 Membrane Product Application Note".

REFERENCE:

"Membrane Handbook", Ed. by W.S.W. Ho and F.Y. Cirkor, Van Nostrand Reinhold, New York, 1991.

WEST**Generate Collection**

114: Entry 1 of 4

File: USPT

Dec 2, 1997

US-PAT-NO: 5699227

DOCUMENT-IDENTIFIER: US 5699227 A

TITLE: Catalyst mediated method of interfacial polymerization on a microporous support, and polymers, fibers, films and membranes made by such method

DATE-ISSUED: December 2, 1997

INVENTOR-INFORMATION

NAME	CITY	STATE	ZIP CODE	COUNTRY
Costa; Lawrence C.	Mansfield	MA		

ASSIGNEE-INFORMATION

NAME	CITY	STATE	ZIP CODE	COUNTRY	TYPE CODE
Ionics, Incorporated	Watertown	MA			02

APPL-NO: - 341496

DATE FILED: November 17, 1994

INT-CL: [6] BOLD 61/30, BOLD 67/30

US-CL-ISSUED: 210/650; 210/490; 210/500.38; 96/14; 95/45; 427/245

US-CL-CURRENT: 210/650; 210/490; 210/500.38; 427/245; 95/45; 96/14

FIELD-OF-SEARCH: 427/245; 427/246; 210/490; 210/500.37; 210/500.38; 210/500.41; 210/650; 210/651; 96/14; 264/45.1; 95/45

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

Search Selected

Search ALL

PAT-NO	PUBL-DATE	PATENTEE NAME	US CL
4244442	July 1981	Smith	210.5
4259181	March 1981	Radette	210.54
4270344	July 1981	Radette	210.54
4287785	August 1981	Jaguillat et al.	428.14
4324774	April 1982	White et al.	424.17
437343	August 1982	Rak et al.	427.44
4413767	October 1982	Kamiyama et al.	210.47
4443323	February 1987	Sundet	210.5.13-
4761234	August 1988	Uemura et al.	210.5.137
4783346	November 1988	Sundet	427.44
4814382	March 1989	Wrasidlo	210.47
4828708	May 1989	Bray	210.584
4831885	May 1989	Tran et al.	427.44
4852122	August 1989	McCrory	210.521.47
4872384	October 1989	Tomaschke	210.5.13-
4948506	August 1990	Lonsdale et al.	210.491
4948537	August 1990	Tomaschke	210.5.13-
4950424	August 1990	Chau	210.5.137
5006628	April 1991	Jackson, Jr. et al.	528.142
5019264	May 1991	Arthur	210.5.137
5051178	September 1991	Uemura et al.	210.5.13-
5084182	January 1992	Arthur	210.442
5085777	February 1992	Arthur	210.5.13-
5152001	October 1992	Hodgdon	210.454
5234598	August 1993	Tran et al.	210.414
5246587	September 1993	Tomaschke	210.654

FOREIGN PATENT DOCUMENTS

FOREIGN-PAT-NO	PUBL-DATE	COUNTRY	US CL
744315A1	November 1981	EPX	
744454A1	July 1981	DEX	
883822A1	January 1988	EPX	
281397	August 1990	DEX	
2152520	December 1991	JPX	
4358537	November 1992	JPX	
WO 92/19065	November 1992	KCX	

OTHER PUBLICATIONS

McCrory, S., Bend Research, Inc., "The Mechanism of Haptogen Antigen Binding to the Reverse Transcription Membrane Phase II", Technical Report, Jan. 1991.

The Handbook of Industrial Membrane Technology, M. J. Porter, Ed., 1987, at 127-128.

Hille, G. et al., Angew. Chem. Int. Ed. Engl. 17, 1047, at 1047.

Smirnov, E., Chemical Society Reviews, vol. 12, No. 2, 1983.

"4-Alkylaminopyridines: Super Acylation Catalysts" at 127-128.

ABSTRACT: 12.

PRIMARY-EXAMINER: Dredge, Joseph W

ATTY-AGENT-FIRM: Lippin & Kuster LLP

ABSTRACT:

The invention pertains to the polymerization of molecules having electrophilic and nucleophilic substituents on the same or different molecules, such polymerization initiated by aminopyridine catalysts which are substantially regenerated during polymerization. The invention also pertains to polymer products of such polymerization, to permselective membranes containing such polymers, and to processes and apparatus for separating fluids.

45 Claims, 2 Drawing figures

WEST[Generate Collection](#)**Search Results - Record(s) 11 through 16 of 16 returned**☐ 11 Document ID: US 6001889 A

LIT: Entry 11 of 16 File: USPT Oct 14, 1999

US-PAT-NO: 6 1889 A

DOCUMENT-IDENTIFIER: US 60 1889 A

TITLE: Polymers with fractal structure

DATE-ISSUED: December 14, 1999

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Lefebvre; Michel S. M.	Kurrajong Heights			AUX

US-CL-CURRENT: 521 52

Full	Title	Citation	Front	Review	Classification	Date	Reference
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KWIC	Draw Desc	Image
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☐ 12 Document ID: US 5968585 A

LIT: Entry 12 of 16 File: USPT Oct 19, 1999

US-PAT-NO: 5968585

DOCUMENT-IDENTIFIER: US 5968585 A

TITLE: Process for recovery of protein from aqueous media in corn wet milling

DATE-ISSUED: October 19, 1999

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Liaw; Gin C.	Deeratur	IL		
Cheryan; Munir	Urbana	IL		

US-CL-CURRENT: 426 656, 210 641, 210 650, 210 651, 210 653, 426 478, 426 488, 426 629, 530 376, 530 377, 530 414

Full	Title	Citation	Front	Review	Classification	Date	Reference
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KWIC	Draw Desc	Image
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☐ 13 Document ID: US 5773076 A

LIT: Entry 13 of 16 File: USPT Jan 31, 1999

US-PAT-NO: 5616595 A
DOCUMENT-IDENTIFIER: US 5616595 A

TITLE: Process for recovery of insoluble protein from steep water

DATE-ISSUED: June 30, 1996

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Liaw; Gin C.	Decatur	IL		
Liaw; Eric H.	Decatur	IL		
Sherry; Martin	Urbana	IL		

US-CL-CURRENT: 428 456, 210 641, 210 650, 210 681, 210 683, 424 428, 424 429, 424 428, 520 276, 520 414

Full	Title	Citation	Front	Review	Classification	Date	Reference
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KWIC	Draw Desc	Image
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14. Document ID: US 5616595 A

L17: Entry 14 of 16

File: USPT

Apr 1, 1997

US-PAT-NO: 5616595

DOCUMENT-IDENTIFIER: US 5616595 A

TITLE: Process for recovering water insoluble compounds from a fermentation by to

DATE-ISSUED: April 1, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Chu; Alexander H. T.	Buffalo Grove	IL		
Wloch; Gene P.	Lake Villa	IL		

US-CL-CURRENT: 514 244

Full	Title	Citation	Front	Review	Classification	Date	Reference
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KWIC	Draw Desc	Image
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15. Document ID: US 5279739 A

L17: Entry 15 of 16

File: USPT

Jan 18, 1994

US-PAT-NO: 5279739

DOCUMENT-IDENTIFIER: US 5279739 A

TITLE: Durable filtration membrane having optimized molecular weight

DATE-ISSUED: January 18, 1994

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Pemawansa; K. P.	Bradford	MA		

US-CL-CURRENT: 210 61141, 210 44, 210 611, 210 612, 210 613, 424 428, 424 429

Full	Title	Citation	Front	Review	Classification	Date	Reference
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KWIC	Draw Desc	Image
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16 Document ID US 5147541 A

117: Entry 14 of 14

File USPT

Sep 18, 1992

US-PAT-NO: 5147541

DOCUMENT-IDENTIFIER: US 5147541 A

TITLE: Spiral filtration module with strengthened relatively flat, and relatively constructing same

DATE ISSUED: September 18, 1992

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
McDermott, Jr., Thomas C.	Lynn	MA		
Skelton, Daniel F.	Billerica	MA		

US-CL-CURRENT: 210 321.74; 156 290, 210 321.83, 210 482.4, 42 157, 42 17, 42 179

Full	Title	Citation	Front	Review	Classification	Date	Reference
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KWIC	Draw Desc	Image
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Terms

Documents

116 and nanofiltration

16

Display

10 Documents, starting with Document: 16

Display Format: CIT Change Format